Exhibit 14.14

United States' Motion to Enter Consent Decree,
United States v. Alden Leeds, Inc. et al., Civil Action No. 22-7326 (D.N.J.)

EXHIBIT A-43

Appendix A to OxyChem's Comments in Opposition to Proposed Consent Decree,
United States v. Alden Leeds, Inc., et al., Civil Action No. 2:22-cv-07326 (D.N.J.)

Dr. W. S. Gump
January 8, 1945

1.
2. ~~[illegible]~~
3. ~~Dr. Gump 6/28/62~~
4. destroyed 9/28/70
~~[illegible] (Geneva) 12/5/68~~

IMPROVED PROCESS FOR THE MANUFACTURE
OF COMPOUND G-11

The large amounts of sulfuric acid needed in the present process for the manufacture of G-11 and the difficulty of the disposal of the waste sulfuric acid made it desirable to find a method where considerably smaller amounts of sulfuric acid would be applied or where the acid could be reused.

In the first experiments, the 93% sulfuric acid was replaced by 62% and 80% acid which could be filtered off from the reaction product and re-used. However, the results in regard to the quality of the G-11 were inferior.

At present, 715 g. of sulfuric acid 93% are employed for 100 g. of trichlorophenol; it was found that this amount of acid could be cut down to one-third without diminishing the yield or changing the quality of the product.

A much bigger reduction of the amount of sulfuric acid used could only be achieved by carrying out the reaction at a temperature high enough to keep the mixture in a liquid state. Aqueous formaldehyde had to be replaced by trioxane or paraformaldehyde. A large number of experiments showed that the optimum temperature lies between 130° and 140°, that the time of reaction should be very short (a few minutes), that oleum 20% which gives slightly better results than 93% sulfuric acid should be used to the amount of 65 g. per one mol (198 g.) of trichlorophenol. An excess of paraformaldehyde (25% above the theoretical amount) must be used in order to re-

duce unchanged trichlorophenol to a minimum. Iron apparatus is entirely satisfactory.

The modified process is illustrated by the following example:

A mixture of 198 g. of Dowicide #2, purified by vacuum distillation, and of 18.8 g. of paraformaldehyde are heated to 75° and well stirred. 65 g. of oleum 20% is added dropwise and the addition is so regulated that the temperature rises slowly without outside heat, reaching about 135° at the end. The addition of the oleum takes 10 to 15 min. The mixture is stirred for two minutes more and then allowed to run into a solution of 100 g. of caustic soda flakes in 1000 cc of water. The reaction flask is washed with a solution of 25 g. of caustic soda flakes in 250 cc of water. The combined alkaline solutions are heated to boiling until practically all of the melt has gone into solution, and are filtered over filter-cel. 6 g. of alkali-insoluble material remain on the filter pad.

Under good stirring, 62% sulfuric acid (about 120 g.) are added dropwise to the alkaline solution until a pH of 10.3 was reached. The G-11 sodium salt formed is filtered off, and washed with 200 cc of water. It is then suspended in 2 l. of water and acidified with sulfuric acid under stirring until congo red paper turns blue; about 30 g. of 62% sulfuric acid are needed. The G-11 Tech. is filtered, washed with water until acid-free and dried. 170 g. (84% of the theory) of the m.p. 154°-158° are obtained.

The alkaline mother liquor of the G-11 sodium salt was acidified and steam distilled. No unreacted Dowicide #2 was recovered; however, 24 g. of resinous material remained.

Yield:	G-11 Tech.	170 g.
	Resin alkali-soluble	24 g.
	Alkali-insoluble	6 g.
	Total	200 g.

W. Gump.

Dr. W. S. Gump
Delawanna, New Jersey
January 8, 1945.